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(54) **Transparent coatings by reactive sputtering.**

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JOURNAL OF THE AMERICAN CERAMIC SOCIETY, vol. 46, no. 4, April 1963, pages 161-167, American Ceramic Society, Columbus, Ohio, US; J.C.WILLIAMS et al.: "Preparation of thin mullite films"

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Description

The invention relates to the cathode sputtering of metals in the presence of a reactive gas to form a durable, transparent coating on a substrate.

Windows of modern buildings and vehicles are provided with coatings having particular optical properties. Thin metal films are deposited on glass or plastic to increase the reflectance of solar radiation. Even more energy efficient windows include a multilayer dielectric-metal-dielectric coating having a high visible transmittance combined with high reflectivity and low emissivity in the infrared wavelength range of the electromagnetic spectrum. The index of refraction of the dielectric layer is preferably 2.0 or greater in order to minimize the visible reflectance and enhance the visible transmittance of the window. Such multilayer coatings are frequently deposited by sputtering metal targets in an atmosphere of inert gas to deposit a metal layer and in an atmosphere containing a reactive gas to deposit the dielectric layers. Such sputtered coatings are described in U.S. Patent 4,610,771 (Gillery).

One such dielectric-metal-dielectric coating comprises successive layers of zinc oxide, silver, zinc and zinc oxide deposited on a float-glass substrate. Silver is preferred because of its high infrared reflectance, but the metal layer may also be gold, copper, or another good electrical conductor. When silver is used, it is usually covered with a very thin layer of zinc, tin, titanium, aluminium or other metal to protect the silver from corrosion. Zinc oxide is a preferred dielectric because it has a refractive index of 2.0 and it can be deposited by reactive sputtering at rates which are relatively high for oxides. Various other materials, such as oxides of bismuth, titanium, tin and indium, and nitrides of aluminium are also used as dielectric layers.

Usually the coated glass is sealed as part of a double-glazed window unit in which the coating lies on an inside surface where the coating is protected by glass from abrasion and environmental materials which could cause corrosion and degrade the optical properties of the coating. Abrasion and corrosion are problems even while handling of the coated glass before and during the fabrication process.

There is a recognized need for a harder and more corrosion resistant dielectric material to provide a protective overcoat for metal or dielectric coatings or to replace some or all of one or both of the dielectric layers in a dielectric-metal-dielectric coating. The present invention relates to the use of reactively sputtered aluminium-silicon alloys for these and other purposes, for example the provision of a coating that reduces transmissivity of IR-radiation through a substrate such as glass or plastics.

Aluminium-silicon alloys have previously been sputtered in a reactive gas.

In a paper "Preparation of Thin Mullite Films", J. Am. Ceram Soc., Vol. 46, pp. 161-167 (1963), J. C. Williams et al, disclosed deposition of mixed Al_2O_3/SiO_2 films by sputtering in oxygen of aluminium-silicon alloy targets having 20, 28 and 40 wt.% silicon.

In a paper, "Influence of Residual Gases on the Properties of DC Magnetron-Sputtered Aluminium-Silicon", J. Vac. Sci. Technol., Vol. 17, pp. 384-387 (1980), R.S. Nowicki disclosed metal films deposited by sputtering a target of aluminium-1.5% silicon in gas of argon-1% oxygen.

According to the present invention there is provided a method for preparing a coated substrate comprising:

depositing a coating on the substrate;

preparing a sputtering target of an alloy comprising aluminium and sufficient silicon to produce an amorphous overcoating;

placing the target and the coated substrate in an evacuated chamber; and

sputtering the target in an atmosphere comprising a gas having components which react with aluminium and silicon so as to form a transparent amorphous overcoating containing reaction products of aluminium and silicon.

The invention also provides articles when coated by this method.

The coating may be a dielectric-metal-dielectric or other coating on a transparent glass or plastic base.

The lower level of silicon which must be present in the alloy to obtain an amorphous (glassy) coating is not precisely known. It is believed that aluminium-2% silicon forms oxide coatings which are primarily crystalline or microcrystalline and which are therefore more permeable and less suitable as corrosion barrier layers. The useful upper limit of silicon is also not precisely known. The electrical conductivity of pure silicon is so low that it is unsuitable for sputtering with direct current. As little as 0.2% aluminium in silicon provides adequate conductivity. Inclusion of about 5% aluminium in silicon would significantly lower the cost of a sputtering target and a target having about 10% aluminium would be likely to have a significantly higher deposition rate. Aluminium-silicon alloys which can be fabricated easily into dense targets suitable for high rate sputtering are preferred. Targets formed by vacuum casting of an alloy of approximately 70% aluminium and 30% silicon were so porous that arcing during reactive sputtering was a severe problem. The phase diagram of aluminium-silicon has a eutectic at 88.3 percent aluminium and 11.7 percent silicon.

Compositions having approximately 88 percent aluminium and 12 percent silicon with incidental impurities are especially preferred because dense, easily sputtered targets can be prepared readily and inexpensively by conventional methods. Alloys containing significant amounts of metal in addition to aluminium and silicon would have eutectics at other concentrations. Taking these factors into consideration, the preferred range for the aluminium-silicon alloys according to the invention is 6 to 18 percent by weight silicon. For binary alloys, the corresponding range is 82 to 94% aluminium. A range of 10 to 14 percent silicon is more preferred.

The invention further provides a method for depositing a transparent coating on a substrate comprising:

preparing a sputtering target of an alloy comprising aluminium and 6 to 18% silicon;

placing the target and the substrate in an evacuated chamber;

sputtering the target in an atmosphere comprising a gas having components which react with aluminium and silicon so as to form an amorphous coating containing reaction products of aluminium and silicon. The invention additionally provides articles created by this method.

The sputtering gas is preferably a mixture of an inert gas, preferably argon, and a reactive gas. Oxygen, nitrogen, mixtures of oxygen and nitrogen, and gaseous compounds of oxygen and nitrogen, such as nitrous oxide, are preferred but other reactive gases are possible. Use of a mixture of argon and oxygen produces films having an index of refraction of approximately 1.6 and a normalized deposition rate (as defined below) of 15 nm (150 Å) mm^2/J. A mixture of argon and nitrogen produces films having an index of 2.2 and a normalized deposition rate of 50 nm (500 Å) mm^2/J. A mixture of argon, oxygen and nitrogen produces films having intermediate values of the refractive index and deposition rate.

The film deposited according to the invention contains reaction products of aluminium and silicon with reactive components from the reactive gas. When the sputtering gas contains oxygen, the deposited coating contains a mixture of aluminium and silicon oxides. When the sputtering gas contains nitrogen, the coating contains a mixture of aluminium and silicon nitrides. All of these four compounds are relatively hard. These reaction products form an amorphous film which is a better barrier against corrosion than zinc oxide and other materials which form polycrystalline films.

The method and coated articles according to the invention provide durable, transparent coatings which can be deposited at relatively high rates utilizing easily sputtered targets having a reasonable cost. The thickness and sometimes the index of refraction of the coatings can be adjusted to obtain tinted or essentially colorless films, as desired. The hardness and corrosion resistance makes the coating less susceptible to environmental conditions and handling while a coated glass is made into a double-glazed window unit.

The invention will now be described by way of example with reference to the accompanying drawings in which Figure 1 is a graph showing the normalized deposition rate D in 0.1 nm mm^2/J (Å mm^2/J) as a function of the ratio r of O_2 flow to the total flow of O_2 and N_2 for coatings deposited by sputtering a target of 88% aluminium and 12% silicon in a mixture of argon, oxygen and nitrogen according to the invention.

Figure 2 is a graph showing the index of refraction as a function of ratio of O_2 flow to the total flow of O_2 and N_2 for coatings deposited by sputtering a target of 88% aluminium and 12% silicon in a mixture of argon, oxygen and nitrogen according to the invention.

Figure 3 is a view of a cross-section of a double-glazed window unit incorporating a transparent sheet coated according to the invention.

The coatings are prepared with conventional sputtering equipment. One or more sputtering sources are placed in a chamber which can be sealed and evacuated by conventional vacuum pumps. If metal and dielectric coatings are to be deposited, the sources may be separated by gates or isolation regions which are separately evacuated to prevent cross-contamination of sputtering gases. The pressure and flow rate of the sputtering gases are also controlled by conventional devices. If a mixed gas is used, the various components are separately controlled. Each sputtering source is connected to an appropriate power source, preferably a direct current source having provision for automatically maintaining the voltage, current or power, as desired.

A first sample was deposited in a vacuum system comprising a load lock and a coating chamber fitted with one round and two rectangular planar magnetron sputtering sources of the type disclosed in U.S. Patent 4,166,018 to Chapin. The round source had a silver target 6 inches in diameter. The two rectangular sources had 102 mm x 203 mm (4 x 8 inch) targets, one of zinc and the other of an aluminium-silicon alloy. The target was 73% aluminium and 27% silicon with incidental impurities and was prepared by the conventional steps of mixing appropriate amounts of metal powder; melting, casting and cooling in vacuum; and then machining to final size. 102 mm x 102 mm or 101 mm x 154 mm (4 x 4 or 4 x 6 inch) glass substrates were cleaned and then coated with 40 to 45 nm (400 to 450 Å (angstroms) of zinc oxide deposited by sputtering a zinc target in an argon-oxygen atmosphere. This was followed by a layer of 4 to

4.5 nm (40 to 45 Å) silver and then approximately 0.5 nm (5 Å) zinc deposited by sputtering the metal targets in an argon atmosphere. Next came another layer of 40 to 45 nm (400 to 450 A) of zinc oxide. This dielectric-metal-dielectric coating was provided with a protective overcoat by reactively sputtering the aluminium-silicon target in a sputtering gas comprising argon and oxygen at an argon:oxygen flow ratio of 20:1 at a total pressure of 1.2 Pa (6 mT or 6 x 10^{-3}Torr). The sputtering potential was 500 V for the deposition of the aluminium-silicon oxide and the zinc oxide, 390 V for silver and 800 V for zinc. The refractive index of the aluminium-silicon oxide layer was 1.57.

The sample coatings of Tables 2 to 6 were made in a Model ILS-1600 sputtering system manufactured by Airco Solar Products. This system has a load lock and a single coating chamber having three separate planar magnetron sputtering sources to which were mounted targets of zinc, silver and the aluminium-silicon alloy. All targets were 127 mm x 432 mm (5 by 17 inches) in size and the aluminium-silicon targets were approximately 6.4 mm (0.25 inches) thick. One target was approximately 72% aluminium with 28% silicon and incidental impurities and a second was approximately 88% aluminium with 12% silicon and incidental impurities. These targets were prepared as described above except that the 12% silicon target was cold-rolled before it was machined. The substrate to be coated was cleaned and placed on a horizontal conveyor system which transported the substrate through the system making one or more passes under the sputtering sources.

Typically, with this system, an 80 A layer of silver was deposited in one pass with a line speed of approximately 57 mm/sec, at a sputtering power of 0.5 kW and a potential of 300 V, in an argon atmosphere at 0.8 Pa (4 mT). A 35 nm (350 Å) layer of zinc oxide was deposited in one pass at a line speed of 15 mm/sec, at 3 kW and 390 V, in an atmosphere containing argon and oxygen with a ratio of 1:15 and a total pressure of 0.6 Pa (3 mT). A 30 nm (300 Å) layer of (88% Al, 12% Si) oxide was typically deposited in one pass with a line speed of approximately 5 mm/sec, at a power of 6.5 kW and potential of 280 to 300 V, in an atmosphere of argon and oxygen with a ratio of 1:2 at a total pressure of 0.6 Pa (3 mT). A 30 nm (300 Å) layer of (88% Al, 12% Si) nitride was typically deposited in one pass at 13 mm/sec, at 6.5 kW and 300 to 320 V, in an atmosphere of argon and nitrogen with a ratio of 1:2 at a total pressure of 0.6 Pa (3 mT). A 30 nm (300 Å) layer of (88% Al, 12% Si) oxynitride was typically deposited in one pass with a line speed of 9 mm/sec, at a power of 6 kW and potential of 320 V, in an atmosphere of argon, nitrogen and oxygen with a ratio of 2:4:1 at a total pressure of 0.6 Pa (3 mT).

The normalized deposition rate D for each layer is calculated

$$D = \frac{sdc}{np}$$

where D is the normalized rate in 0.1 nm.mm^2/J (A mm^2/J);
s is the speed of the moving substrate in mm/sec;
d is the thickness of the deposited layer in units of 0.1 nm (Å);
c is the length of the closed-loop erosion region of the planar magnetron sputtering source in mm;
n is the number of times the substrate passes the sputtering source during the deposition; and
p is the sputter power in W.

The values of D for several oxides and nitrides are given in Table 1.

TABLE 1

Compound	ZnO	SnO_2	Al_2O_3	TiO_2	TiN	AlN
D	1500	1000	200	100	380	650
Compound	(88Al, 12Si)N_x		(72Al, 28Si)O_x		(88Al,12Si)O_x	
D	500		200		150	

The sample coatings were subjected to five tests to determine their corrosion resistance and hardness and the numerical results of the five tests were added to obtain a composite durability index C whose range is 4 to 43.

Very briefly, the corrosion testing procedures were as follows:

DI24:

The coating was placed in contact with a square of filter paper which had been saturated with de-ionized water. After 24 hours, the filter paper was removed, the sample blown dry and then photographed under a microscope at 100X and 400X magnification. The photographs were subjectively matched with a scale where 10 is not corroded and 1 is completely corroded.

NaCl24:

The test procedure was the same as for DI24 except that the filter paper was saturated with a solution of 1% by wt sodium chloride in de-ionized water.

Fingerprint:

Fingerprints were placed on sample coatings. After 24 hours, and again after 2 weeks, the coatings were cleaned with Kimwipes tissues and conventional glass cleaner. The samples were evaluated against a scale where 3 represented no corrosion, 2 a slight stain or other effect and 1 represented visible corrosion.

The hardness of the films was evaluated by two tests:

Eraser Tests:

A coating was rubbed five times with a conventional pencil eraser and photographed under a microscope at 50X magnification. The photographs were matched to a scale where 10 represented no effect and 1 indicated that most of the film had been rubbed off. This test was performed twice for each sample.

Taber Abrader:

102 mm x 102 mm (Four by four inch) sample coatings were abraded by 50 revolutions of CS-10F wheels with 500 g applied mass. The wheels were resurfaced for 25 revolutions after every 10 revolutions of abrasion. After 50 revolutions, the samples were photographed at 50X magnification and the number of scratches in a typical 25 mm by 25 mm (one by one inch) square was determined. A score was computed as 10 - 9N/50 where N is the number of scratches. On this scale, zero scratches scores a 10, 50 scratches scores a one and greater than 50 scratches is assigned a score of 0.

The optical properties of the samples were determined with a spectrophotometer. Y_F is the film side reflectance integrated over the visible spectrum as represented by the intensity parameter on the CIE chromaticity scale. Y_G is a similar parameter for the glass side reflectance, and Y_T is the visable transmittance of the sample. In some cases, the emissivity e and the sheet resistance R_s (ohms/square) were measured.

The samples of Tables 2 and 3 include a dielectric-metal-dielectric coating. This coating is referred to as the ZnO Reference coating and has the nominal composition (35 nm/350 Å) ZnO + (8 nm/80Å) Ag + (2 nm/20 Å) Zn + (52 nm/520 Å) ZnO. The durability and optical properties for a typical sample of this coating are given in the first row of Table 5 and Table 6.

Table 2 shows the composition of the aluminium-silicon alloy target, the reactive gas R, ratio r of O_2 flow to the total flow of O_2 and N_2, the overcoating thickness t, and the composite durability factor C for a group of coatings where a dielectric-metal-dielectric coating was overlayed with a protective coating deposited by reactively sputtering an aluminium-silicon alloy.

TABLE 2

Glass + D-M-D coating + (Al,Si)R_x overlayer where D-M-D is the ZnO Reference coating					
Sample	%Si	Overlayer			
		R gas	t(nm)	C	r
13-1	12	N_2	30	34	0
12-1	12	O_2	30	33	1
94-1	12	N_2	30	37	0
113	12	$O_2 + N_2$	30	39	0.3
72-2	28	$O_2 + N_2$	30	34	0.2

Table 3 illustrates the aluminium-silicon composition, the reactive gas R, the layer thickness t, and composite durability factor C for a group of samples where a dielectric-metal-dielectric coating was provided with both an underlayer and an overlayer deposited by reactive sputtering of an aluminium-silicon alloy.

TABLE 3

Glass + (Al,Si)R_x underlayer + D-M-D coating + (Al,Si)R_x overlayer where D-M-D is the ZnO Reference coating						
Sample	%Si	Underlayer		Overlayer		C
		R gas	t(nm)	R gas	t(nm)	
14-2	12	N_2	30	N_2	30	35
15-2	12	O_2	30	N_2	45	37
12-3	12	O_2	30	O_2	30	-
26-1	28	O_2	30	O_2	30	34

Table 4 is similar to Table 3 but shows the parameters for a group of samples where a silver coating was provided with an underlayer and an overlayer of a reactively sputtered aluminium-silicon alloy.

TABLE 4

Glass + (Al,Si)R underlayer + Ag(8 nm) coating + (Al,Si)R overlayer								
Sample	%Si	Underlayer			Overlayer			C
		R gas	r	t(nm)	R gas	r	t(nm)	
53-1	28	N_2	0	36	N_2	0	55	24
68-1	28	O_2	1	52	O_2	1	75	20
73-1	28	$O_2 + N_2$	0.2	25.3	$O_2 + N_2$	0.2	37	23

Table 5 shows the results of the individual corrosion and hardness tests of a ZnO Reference coating (Sample 29-1) and a group of glass coatings made in accordance with the invention. The composition of the remaining samples in Table 5 is given in one of the Tables 2 to 4.

TABLE 5

Durability Test Data						
Sample	DI24	NaCl24	Fingerprint	Eraser	Taber	Composite
29-1	6	6	1	6	6	25
13-1	7	7	3	9	8	34
12-1	9	9	3	6	6	33
94-1	10	8	3	8	8	37
113	10	10	3	7	9	39
72-2	9	6	2	9	8	34
14-2	9	8	3	8	7	35
15-2	9	7	2	10	9	37
12-3	-	-	3	7	-	-
26-1	8	7	3	9	8	34
53-1	9	4	3	8	0	24
68-1	4	6	2	8	0	20
73-1	4	3	3	8	5	23

Table 6 shows the optical properties of the ZnO Reference coating (Sample 29-1) and a group of sample coatings made according to the invention. The coating composition of each of the other samples of Table 6 is given in one of Tables 2 to 4. The emissivity of the ZnO Reference coating is 0.1. Experience indicates that a dielectric-metal-dielectric coating having a sheet resistance R_s of 10 ohms per square or less will have an emissivity of 0.1 or less.

TABLE 6

Optical Test Data				
Sample	Y_F	Y_G	Y_T	R_s
29-1	8	11	80	8
13-1	29	28	64	9
12-1	13	15	79	7
94-1	6	9	83	9
113	5	9	83	10
72-2	4	10	76	-
14-2	-	-	-	9
15-2	-	-	-	7
12-3	-	-	77	7
26-1	19	-	68	-
53-1	9	11	84	10
68-1	10	12	81	-

Figure 1 shows the normalized deposition rate D as a function of the ratio r of oxygen to the total of oxygen and nitrogen for a group of coatings of (88% Al, 12% Si) oxynitride on glass. The argon:nitrogen ratio was constant at 1:2. The numbers indicated next to the data points are the atomic percentage of oxygen in the films as measured by electron spectroscopy for chemical analysis (ESCA).

Figure 2 shows the index of refraction for a group of coatings similar to those of Figure 1 except the argon:nitrogen ratio was 3:7.

The numbers indicated next to the data points in Figure 2 are scores on a scale of 1 (peeling) to 10 (no noticable effect) based on exposure of 24 hours in a cabinet at 120 ° F (49 ° C) at a relative humidity of 95 to 100%. These data were for 30 nm (300 Å) layers of (88% Al, 12% Si) oxynitride applied over a coating similar to the ZnO Reference cooling except that the second ZnO layer was only 22 nm 220 Å thick.

Figure 1 indicates that the deposition rate drops rapidly as the oxygen flow ratio r increases up to about 0.3. Figure 2 indicates that the index of refraction decreases for r greater than 0.1 but that the humidity resistance increases rapidly until r exceeds to about 0.2. The range for r from about 0.1 to 0.3 is preferred.

Figure 3 shows a view of a cross-section of a double-glazed window unit. The unit comprises two transparent sheets 1 and 2 separated by a gap which is bridged by a conventional hermetic seal 3 around the aligned edges of the sheets. The sheets can be glass, plastic or other suitable material. Sheet 1 is the base for a coating 4, which may be a dielectric-metal-dielectric coating, and which is covered over by an amorphous coating 5 in accordance with the invention.

In this description and the claims all percentages of alloy constituents are percentages by weight.

Claims

1. A transparent article comprising a substrate, a coating on the substrate, and a protective overcoating on the coating, wherein the overcoating comprises an amorphous dielectric layer of reaction products formed by sputtering in a reactive gas an alloy target comprising aluminium and silicon.

2. An article according to Claim 1 in which the alloy target contains 6 to 95% silicon.

3. An article according to Claim 1 or Claim 2 in which the coating comprises a multi-layer coating of a dielectric layer/metal layer/dielectric layer and the materials of the dielectric layers are selected from oxides of bismuth, indium, tin, titanium, and zinc.

4. An article according to Claim 3 in which at least one dielectric layer is of zinc oxide.

5. A coated article according to Claim 1 or Claim 2 in which the coating comprises a dielectric layer and a metal layer.

6. A coated article according to anyone of Claims 3 to 5 in which the metal layer comprises silver.

7. A coated article according to any preceding claim in which the alloy target contains 6 to 18% of silicon.

8. An article according to any preceding claim in which the reactive gas is selected from oxygen, nitrogen, compounds of oxygen and compounds of nitrogen.

9. An article according to Claim 8 in which the reactive gas contains oxygen and nitrogen and the ratio of oxygen to the total of oxygen and nitrogen is in the range 0.1 to 0.3.

10. An article according to any preceding claim in which the alloy target comprises 10 to 14% silicon and 86 to 90% aluminium.

11. An article according to Claim 10 in which the alloy target comprises 88% aluminium, 12% silicon, excluding incidental impurities.

12. A transparent article comprising a substrate and an amorphous coating of reaction products formed by sputtering in a reactive gas an alloy target comprising aluminium and from 6 to 18% silicon.

13. A method for preparing an article of Claims 1 to 11 comprising:
depositing the coating on the substrate;
preparing a sputtering target of an alloy comprising aluminium and silicon;
placing the alloy target and the coated substrate in an evacuated chamber; and
sputtering the target in an atmosphere comprising a gas having components which react with aluminium and silicon so as to form a transparent amorphous dielectric overcoating containing reaction products of aluminium and silicon.

14. A method according to Claim 13 in which the alloy target contains from 6% to less than 95% silicon.

15. A method according to Claim 14 in which the alloy target contains from 6 to 18% silicon.

16. A method according to Claim 15 in which the alloy comprises 86 to 90% aluminium and 10 to 14% silicon excluding incidental impurities.

17. The method according to Claim 16 in which the alloy comprises 88% aluminium and 12% silicon excluding incidental impurities.

18. A method according to any one of Claims 13 to 17 in which the reactive gas is selected from nitrogen, oxygen, compounds of oxygen and compounds of nitrogen.

19. A method according to Claim 18 in which the reactive gas comprises oxygen and nitrogen and the ratio of oxygen to the total of oxygen and nitrogen is in the range 0.1 to 0.3.

Patentansprüche

1. Transparenter Gegenstand, bestehend aus einem Substrat, einem Überzug auf dem Substrat, und einem schützenden Decküberzug auf dem Überzug, wobei der Decküberzug eine amorphe dielektrische Schicht aus Reaktionsprodukten enthält, die durch Zerstäubung eines Aluminium und Silizium enthaltenden Legierungs-Targets in einem reaktiven Gas gebildet werden.

2. Gegenstand nach Anspruch 1, wobei das Legierungs-Target 6 bis 95% Silizium enthält.

3. Gegenstand nach Anspruch 1 oder 2, wobei der Überzug ein Mehrschichtüberzug aus einer Dielektrikumschicht/Metallschicht/Dielektrikumschicht-Kombination ist und die Materialien der Dielektrikumschichten aus Oxiden von Wismut, Indium, Zinn, Titan und Zink ausgewählt sind.

4. Gegenstand nach Anspruch 3, wobei mindestens eine Dielektriumschicht aus Zinkoxyd besteht.

5. Beschichteter Gegenstand nach Anspruch 1 oder 2, wobei der Überzug eine Dielektrikumschicht und eine Metallschicht enthält.

6. Beschichteter Gegenstand nach einem der Ansprüche 3 bis 5, wobei die Metallschicht aus Silber besteht.

7. Beschichteter Gegenstand nach einem der vorhergehenden Ansprüche, wobei das Legierungs-Target 6 bis 18% Silizium enthält.

8. Gegenstand nach einem der vorhergehenden Ansprüche, wobei das reaktive Gas aus Sauerstoff, Stickstoff, Sauerstoffverbindungen und Stickstoffverbindungen ausgewählt ist.

9. Gegenstand nach Anspruch 8, wobei das reaktive Gas Sauerstoff und Stickstoff enthält und das Verhältnis von Sauerstoff zur Gesamtmenge von Sauerstoff und Stickstoff im Bereich von 0,1 bis 0,3 liegt.

10. Gegenstand nach einem der vorhergehenden Ansprüche, wobei das Legierungs-Target 10 bis 14% Silizium und 86 bis 90% Aluminium enthält.

11. Gegenstand nach Anspruch 10, wobei das Legierungs-Target 88% Aluminium und 12% Silizium, unwesentliche Verunreinigungen nicht berücksichtigt, enthält.

12. Transparenter Gegenstand, bestehend aus einem Substrat und einem amorphen Überzug aus Reaktionsprodukten, die durch Zerstäuben eines Legierungs-Target aus Aluminium und 6 bis 18% Silizium in einem reaktiven Gas gebildet werden.

13. Verfahren zur Herstellung eines Gegenstands nach einem der Ansprüche 1 bis 11, mit:
Aufbringen des Überzugs auf das Substrat,
Herstellen eines Zerstäubungs-Target aus einer Aluminium und Silizium enthaltenden Legierung,
Plazieren des Legierungs-Target und des beschichteten Substrats in einer evakuierten Kammer, und
Zerstäuben des Target in einer Atmosphäre, die aus einem Gas mit Komponenten besteht, welche mit Aluminium und Silizium so reagieren, daß ein transparenter amorpher dielektrischer Decküberzug gebildet wird, der Reaktionsprodukte von Aluminium und Silizium enthält.

14. Verfahren nach Anspruch 13, bei welchem das Legierungs-Target 6 bis weniger als 95% Silizium enthält.

15. Verfahren nach Anspruch 14, bei welchem das Legierungs-Target 6 bis 18% Silizium enthält.

16. Verfahren nach Anspruch 15, bei welchem die Legierung 86 bis 90% Aluminium und 10 bis 14% Silizium enthält, unwesentliche Verunreinigungen nicht berücksichtigt.

17. Verfahren nach Anspruch 16, wobei die Legierung 88% Aluminium und 12% Silizium enthält, unwesentliche Verunreinigungen nicht berücksichtigt.

18. Verfahren nach einem der Ansprüche 13 bis 17, wobei das reaktive Gas aus Stickstoff, Sauerstoff, Sauerstoffverbindungen und Stickstoffverbindungen ausgewählt ist.

19. Verfahren nach Anspruch 18, wobei das reaktive Gas Sauerstoff und Stickstoff enthält und das Verhältnis von Sauerstoff zur Gesamtmenge von Sauerstoff und Stickstoff im Bereich von 0,1 bis 0,3 liegt.

Revendications

1. Article transparent comprenant un substrat, un revêtement sur le substrat, et un sur-revêtement protecteur sur le revêtement, dans lequel le sur-revêtement comprend une couche diélectrique amorphe de produits de réaction formés par pulvérisation électrique, dans un gaz réactif, d'une cible formée en un alliage comprenant de l'aluminium et du silicium.

2. Article selon la revendication 1, dans lequel la cible en alliage contient 6 à 95 % de silicium.

3. Article selon la revendication 1 ou la revendication 2, dans lequel le revêtement comprend un revêtement multicouche formé d'une couche diélectrique/couche de métal/couche diélectrique, et les matières des couches diélectriques sont choisies parmi des oxydes de bismuth, d'indium, d'étain, de titane et de zinc.

4. Article selon la revendication 3, dans lequel au moins une couche diélectrique est en oxyde de zinc.

5. Article revêtu selon la revendication 1 ou la revendication 2, dans lequel le revêtement comprend une couche diélectrique et une couche de métal.

6. Article revêtu selon l'une quelconque des revendications 3 à 5, dans lequel la couche de métal comprend de l'argent.

7. Article revêtu selon l'une quelconque des revendications précédentes, dans lequel la cible en alliage contient 6 à 18 % de silicium.

8. Article selon l'une quelconque des revendications précédentes, dans le cas duquel le gaz réactif est choisi parmi l'oxygène, l'azote, des composés de l'oxygène et des composés de l'azote.

9. Article selon la revendication 8, dans le cas duquel le gaz réactif contient de l'oxygène et de l'azote, et le rapport entre l'oxygène et le total de l'oxygène et de l'azote se situe entre 0,1 et 0,3.

10. Article selon l'une quelconque des revendications précédentes, dans lequel la cible en alliage comprend 10 à 14 % de silicium et 86 à 90 % d'aluminium.

11. Article selon la revendication 10, dans le cas duquel la cible en alliage comprend 88 % d'aluminium, 12 % de silicium, à l'exclusion des impuretés incidentes ou inhérentes.

12. Article transparent comprenant un substrat et un revêtement amorphe en des produits de réaction formés par pulvérisation électrique, dans un gaz réactif, d'une cible formée d'un alliage comprenant de l'aluminium et de 6 à 18 % de silicium.

13. Procédé pour préparer un article selon les revendications 1 à 11, comprenant :

le dépôt du revêtement sur le substrat ;

la préparation d'une cible pour pulvérisation électrique, cible formée d'un alliage comprenant de l'aluminium et du silicium ;

le placement de la cible en alliage et du substrat revêtu, dans une enceinte dont l'intérieur a été mis sous dépression ; et

la pulvérisation électrique de la cible dans une atmosphère comprenant un gaz ayant des constituants qui réagissent avec l'aluminium et le silicium de façon à former un sur-revêtement diélectrique amorphe transparent contenant des produits de la réaction de l'aluminium et du silicium.

14. Procédé selon la revendication 13, dans lequel la cible en alliage contient de 6 % à moins de 95 % de silicium.

15. Procédé selon la revendication 14, dans lequel la cible en alliage contient de 6 à 18 % de silicium.

16. Procédé selon la revendication 15, dans lequel l'alliage comprend 86 à 90 % d'aluminium et 10 à 14 % de silicium, quand on exclut les impuretés incidentes ou inhérentes.

17. Procédé selon la revendication 16, dans lequel l'alliage comprend 88 % d'aluminium et 12 % de silicium, quand on exclut les impuretés inhérentes ou accidentelles.

18. Procédé selon l'une quelconque des revendications 13 à 17, dans lequel le gaz réactif est choisi parmi l'azote, l'oxygène, des composés de l'oxygène et des composés de l'azote.

19. Procédé selon la revendication 18, dans lequel le gaz réactif comprend de l'oxygène et de l'azote, et le rapport de l'oxygène au total de l'oxygène et de l'azote se situe entre 0,1 et 0,3.

FIG. 1

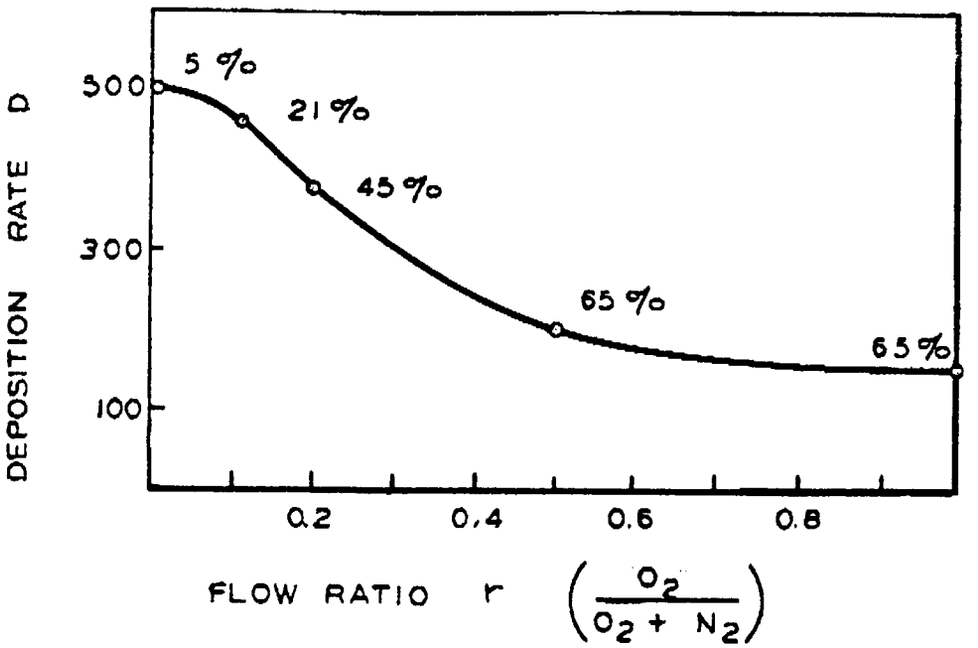

FIG. 2

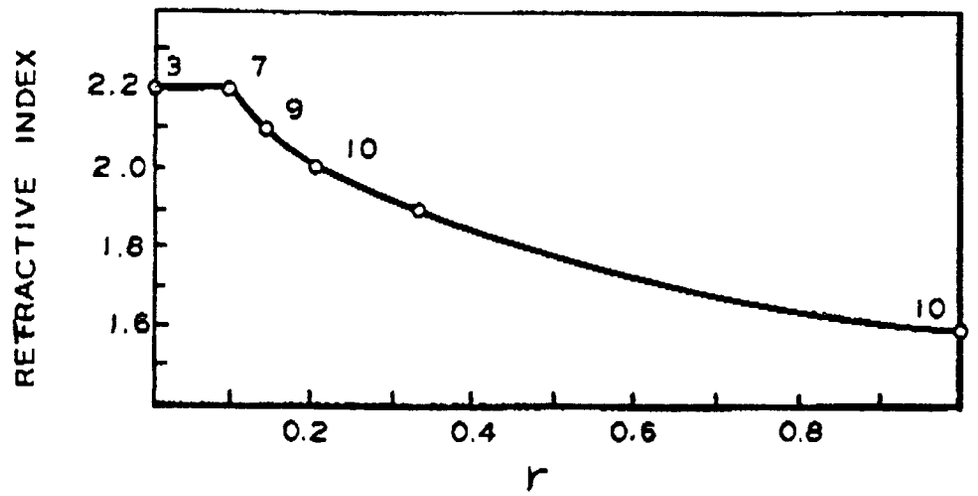

FIG. 3